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(12) The description of the invention
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(56) **1.** The report on a research work, № of state rec. 016 50072107, Kalinin, KPI, 1985, pp. 26-28
2. The inventor's certificate of the USSR № 159005, cl. C 03 B 17/06, 1963.

(54) The method of applying metal coatings on large-size substrates in vacuum and installation for its realization.

(57) Application: this invention deals with optical instrument making, laser technology, electronic and glass industry. Essence of the invention: to apply coating on large-size substrate by known method, including substrate cleaning by inert gas ions and depositing coating simultaneously with ion bombardment of substrate. Ion beam is obtained in accelerator with a selfcontained drift, directed on the substrate at an angle of 15-45 degrees with a surface towards moving substrate at (1,3-5,3)10^{-2} Pa and ion energy of 50-150 eV. Coating deposition begins before the ending of cleaning. Vacuum unit, operating according this method, contains at least two accelerators with a selfcontained drift and plasma generator of depositing metal in the form of tubular cathode with anode encompassing it on a spiral path.
2 for., 3 fig.

The invention deals with optical instrument making, laser technology, electronic and glass industry and can be use for depositing reflecting and other metal coatings on large-size substrates.

Ion plasma methods of depositing metal coatings are known, that include the following preparatory operations – sand blasting, sink, drying, cleaning of processed surface from adsorbed gases by inert gas ion bombardment and coating deposition by it condensation on the processed surface when the arc evaporator is operating. (Investigation and developing of ion plasma coating deposition methods on heating elements. The report on a research work, N 01.85.0014068, Kiev, КАДИ, 1986, pp. 55-56)

Disadvantages of this method are low adhesion, wear resistance and ununiform coating deposition on a large-size substrates, and also a lot of preparatory operations of processed surface.

Methods of coatings applying including an ion-clearing process by a stream of ions and coating by condensation of superimposed matter from gas-and-metal plasma with a simultaneous bombardment of a working surface by ions of sprayed metal and reactionary gas are known.

(To elaborate methods of reliability increase of pneumosystems devices with usage of antifriction coatings. To complete technology of applying of antifriction coatings by ionic - plasma spraying. The report on a research work, N 01850072107, Kalinin, КПИ, 1985, pp. 26-28).

This method provides higher adhesion and wear resistance than previous method, but insufficient for large-size substrates uniformity of coating.

The installation for obtaining coatings in a vacuum, consisting from source of metal plasma including blocks of generation and acceleration of plasma, evacuated chamber, system of a vacuum pumping-out, management system and power supply, mechanism of fastening and travel of parts, and also haulage system of jet gas (Barvinok V.A.Control of stress state and properties of plasma coatings. Moscow, 1990, p. 39) is known.

Disadvantage of this installation is the impossibility of obtaining of uniform coating with a high adhesion (as do not yield an ion-clearing process before applying) on large-size substrates and their large amount for one fill in the chamber.

Most close to the invention on technical substance and achievable technical result is the vacuum installation for metallizing of articles including the camera, system of a vacuum pumping-out, gas-discharge device for clearing of substrates and device for coating as the evaporator of metal set on the reverse movable frame moved between metallized articles, set in the container (A. C. N 159005, cl C 03 C 17/08, 1963).

By the basic disadvantages of described methods of coating applying and installations for it on large-size substrates with a high adhesion, wear resistance and uniformity on all surface of a substrate.

The problem of creation of metal coating applying method on large-size substrates in a vacuum and installation for it ensuring obtaining coatings with high adhesion of coatings to a substrate, wear resistance and uniformity and also increase of installation productivity is the basis of the invention.

The represented problem is solved in such a way that in a known method of metal coatings applying including clearing of a substrate by inert gas ion beam and coating a substrate by cathode spraying in the discharge of inert gas at a simultaneous bombardment of a substrate by

inert gas ion beam. Ion beam is obtained in accelerator with a selfcontained drift (ASD), directed on the substrate at an angle of 15-45 degrees with a surface towards moving substrate at (1,3-5,3)10^{-2} Pa and ion energy of 50-150 eV. Coating deposition begins before the ending of clearing.

By represented method substrates without a composite surface preparation are placed in an evacuated chamber. ASD is switched on and clearing of a substrate surface from impurities, adsorbed gases and from molecules of matter of a substrate having a loose bonding with substrate surface is performed by accelerated stream of ions. The ion-clearing process completely cleans substrate surface up to the «basic» material, thus there is additional polish of substrate surface by ions of inert gas having energy, sufficient for a pulverization of a material of a substrate. A direction of inert gas ion beam at an angle of 15-45^0 to a surface of a substrate will allow most effectively «to blow off» impurities and adsorbed gases. This process is most effective at angle from 45^o up to 0^o. But at angle less than 15^o the process of pulverization of the base material goes inefficiently and there is an effect of polishing substrate material, and also substrate surface clearing goes nonuniformly because of large area of covering. Except for described above processes during the bombardment of a substrate by inert gas ions heating of substrate surface and its activation take place, i.e. molecules and atoms of a substrate transfer in an exited state, that promotes their best bonding with molecules of a coating.

Thus the layer of chemically bound molecules and atoms of the substrate with molecules and atoms of the coating will be formed between a coating and substrate material. The direction of the inert gas ions beam towards to the moving substrate at the angle of 15-45^o allows to treat substrate surface which is located in the working area and the following substrate, located above in a packet in the charge area, that allows to mate a purification process and coating of the first substrate in a working area with cleaning and preparation for coating of the second substrate which is in the charge area. And also it allows to treat large-size substrates with linear dimensions superior the reference linear size of ion beam of ASD.

Cleaning and coating at pressure (1,3-5,3) x 10^{-2} Pa allow to carry out clearing, surface preparation of the substrate and coating most effectively. At pressure lower than 1,3 x 10^{-2} Pa the process of a pulverization of the cathode of plasma generator of superimposed metal (PGSM) in environment of inert gas goes inefficiently . At pressure greater, than 1,3 x 10^{-2} Pa the ions of metal before deposition to the substrate surface, make repeated collisions with ions of inert gas and are implanted in substrate surface from different directions and under

different angles, i.e. they are diffused above a surface uniformly. It gives major uniformity of coating both in properties, and in thickness.

At pressure higher than 5,3 x 10^{-2} Pa there appears the capture of attendant molecules of gas which are present in the camera by a coating surface, and it conducts to deterioration of coating quality, and also the processes of collision in plasma become dominating, so both purification process and coating become inefficient. Also at pressure higher than 5,3 x 10^{-2} Pa the generation and acceleration of inert gas ions in ASD becomes inefficient.

Simultaneous with a coating the bombardment of a substrate by inert gas ion beam allows to maintain molecules and atoms of a substrate in an exited state, as the activation effect drives in time much smaller, than time of applying the coating. Besides, the accelerated ions of inert gas beat out or spray molecules of substrate material which have not strong bond with substrate surface, and also molecule superimposed metal coatings having loose coupling with remaining molecules of the substrate and coating. Thus, on the substrate surface there are only molecules of coating having a strong bond with the substrate and, accordingly, the high adhesion of coating to the substrate and wear resistance of coating is gained.

At energy of inert gas ions less than 50 eV cleaning of substrate surface from gases and films of impurities, chemically bound with it, and also the activation of substrate surface go inefficiently. And at energy more than 150 eV processes of a pulverization of a coating material go more intensively, than process of a spraying. Therefore energy of inert gas ions is selected in the range of 50-150 eV. The inert gas ion beam with such energy is obtained in accelerators with the selfcontained electron drift most effectively. In the given range of energies they have major efficiency, resource, than the known ion accelerators, and differ by a relative simplicity and good regulation of output characteristics, including the energy of accelerated ions. The beginning of coating applying before ending of purification process allows to maintain additional effect of activation obtained at cleaning prior to the beginning of coating applying, and also does not allow a film of gas to adsorb on a cleaned surface up to the coating.

The known vacuum installation for drawing coats contains an evacuated chamber, vacuum-pumping system, mechanism of travel of substrates trimmed in a packet, gas-discharge device as a plasma source of inert gas ions and device for coating applying at the expense of transpiration of metal during heating by an electric current and its deposition.

According to the invention in vacuum installation the plasma radiant of ions is made as, at any rate, two ASD, directed to the exit

section towards to the moving substrate at the angle of 15-45° to a plane of a substrate and set along a line, perpendicular to the traffic direction of the substrate and parallel to the substrate.

PGSM is made as the tubular cathode from superimposed metal and the tubular anode encompassing it on a spiral path electrically isolated from the cathode, joint with a radiant of a spark-over voltage and with a cooling system, thus the cathode is arranged parallel to the substrate. ASD and PGSM are arranged sequentially in traffic direction of the substrate and set on a blanket frame, joint with the mechanism of a vertical displacement. The PGSM cathode is set above the treated substrate apart no more than 300 mms from its surface.

The manufacture of inert gas ion plasma source as ASD allows to receive a well regulated stream of ions with given ion energy 50-150 eV.

ASD now is the most developed source having the greatest resource and reliability. ASD can work on such gases as argon, oxygen, xenon and it dilates technological opportunities of installation. The installation, at any rate, of two ASD on a line, perpendicular motion of a substrate and parallel it allows to receive uniformity of ion current allocation in a plane, perpendicular to the traffic direction of the substrate.

The allocation of an ion current in the jet of plasma accelerators including ASD has hump-shaped character, i.e. maximum at center and minimums on edges. The addition of two or more jets of ASD allows to receive reasonable uniformity at the substrate sizes larger, than reference size of ASD jet. The uniform motion of the substrate under the ion source allows to receive uniformity of a surface preparation in traffic direction. The installation of ASD by exit section towards to moving substrate at the angle of 15-45° allows to supply optimum requirements for cleaning, preparation and activation of substrate surface. Besides it allows to treat the surface of the first substrate which is in a working area and the second substrate, following it in the charge area, simultaneously thus cleaning and preparing it for coating applying, and also to make the working zone of a jet affecting on substrates larger, than its reference size.

The manufacture of PGSM as the tubular cathode and encompassing it on a spiral path anode allows to receive a circular uniform distribution of a sprayed metal plasma stream of the cathode in a plane, perpendicular to axis of the cathode. And the location it parallel to substrate allows to receive uniformity in a direction, perpendicular to the substrate motion Hereby the PGSM length exceeds the size of the substrate in the indicated direction. The construction of PGSM allows to connect it to a cooling system for pick up of a heat obtained at combustion of the low-voltage discharge between the cathode and the anode in inert gas environment.

The location of ASD for obtaining inert gas ions and PGSM for applying metal in substrate traffic direction sequentially and on a blanket frame allows to conduct at first cleaning of the substrate, then to mate bombardment of substrate by inert gas ion beam and coating and to superimpose a plating without an ion bombardment in a continuous mode without interruption between operations.

Varying distance between ASD and PGSM lengthwise, and arrangement reflecting screens above PGSM for change of metal plasma jet helps to achieve that the moving substrate firstly intercrosses the ion beam of ASD, then appears in the zone, where the ion jet of ASD and metal plasma jet of PGSM intercross and there is a metal spraying at the simultaneous bombardment of the substrate by inert gas ions, and in further fell in the area, where there is only metal spraying.

The location of the PGSM cathode on the treated substrate apart no more than 0.3 m from the surface allows to receive energy and metal ion density sufficient for an intrusion in substrate surface, to obtain good adhesion, durability, and also high speed of coating. Offered PGSM, having larger energy of metal ions than ions in evaporators, provides better bond strength of coating with the substrate. Having smaller velocity of metal pulverization from the cathode than in magnetron systems and, accordingly, smaller velocity of coating, it provides the uniformity of coating on large-size substrates unapproachable to magnetron systems and the reasonable productivity is provided with larger working width of PGSM and larger power put in the discharge. Linking of the blanket frame, on which ASD and PGSM with the mechanism of a vertical displacement are arranged allows to supply constant optimum distance no more than 0.3 m between the PGSM cathode and substrate surface at series processing of substrates trimmed in the packet. Each subsequent substrate is below previous, and the mechanism of the vertical displacement lowers the frame with ASD and PGSM to the quantity, equal to distance between substrates. The mechanism of a vertical displacement can be made as a pantograph, joint with a drive unit.

The substance of the invention is illustrated by drawings. Fig. 1 shows the vacuum. Fig. 2 shows the location of ASD, PGSM and substrate in the cabinet, plan view, and also allocation of an ionic current in the jet of every ASD separately and allocation obtained at cross and addition of two ASD jets. Fig. 3 shows the location of ASD and PGSM concerning the substrate, outboard profile, different zones of treatment formed as a result of ASD and PGSM jets cross.

The vacuum installation (fig. 1) contains the cabinet 1, system a pumping-out 2, mechanism of substrate travel 3, made as a duct 4, in which the underpans with substrates 6, trimmed in a packet in the charge

zone on guiding 5 and joint through two racks 7 with trips with the drive device 8 move, containing the drive (is not shown), ASD 9 and PGSM 10, set on a blanket frame 11, joint with the mechanism of a vertical displacement 12, set on the cabinet 1, the electrical power systems of ASD and PGSM and PGSM cooling (are not shown). The allocation of an ion current in one jet of ASD 9 is shown on the profile 13 (fig. 2), and at cross and addition of two jets - on the profile 14. The zones of treatment formed at cross of ASD15 jets and of PGSM 16 metal plasma jets (fig. 3) are listed herein:

I - the charge zone, where preliminary cleaning and substrate surface preparation take place;

II - the working area, where inert gas ion bombardment and metal coating spraying take place simultaneously;

III – the area of spraying;

IV -- the zone of warehousing of treated substrates.

Fig. 3 shows the substrates in different zones of treatment: 17 - in the charge zone, 18 - in a working area, 19 - in the zone of warehousing.

The method is realized as follows. At the beginning of the process the system a pumping-out creates in an evacuated chamber residual pressure 6.7×10^{-3} Pa. The current supply and rate of argon flux to ASD are switched on, the discharge ignites and surface purification process and its preparation for coating spraying begin.

The current supply and water cooling to PGSM is switched on, the discharge between the anode and cathode ignites, and the process of a spraying starts. Regulating delivery of an argon in ASD, pressure $(1,3\text{-}5,3) \times 10^{-2}$ Pa is set in the cabinet. With the help of regulation of ASD electric power supply voltage energy of inert gas ions in the range of 50-150 eV. The mechanism of substrate travel to the working area and the zone of warehousing is switched on. The substrate goes from the charge area to the working area and further to the zone of warehousing uniformly. At that time the substrate which is in the zone I, is previously decontaminated under action of ion beam formed by ASD and directed at the angle of 15-45° to substrate surface. For regulation and the obtaining of a uniform distribution of inert gas ion current on a substrate axis of ASD can equally revolved in a plane, parallel to the plane of the substrate, but thus ion beams are always guided towards to moving substrate. Having crossed the zone I, the substrate appears in the zone II, where additional to ions of inert gas ions of metal from gas-and metal plasma generated by PGSM at the expense of a pulverization of the cathode start to fall on the substrate. The voltage between the anode and cathode of PGSM is set depending on desirable thickness of coating, material of the cathode and substrate. Applying of metal coating on the substrate at simultaneous inert gas ion bombardment goes on. Thus the

coating starts at once after surface cleaning and preparation without interruption at the expense of continuous motion of the substrate.

In the further substrate crosses the zone III, and there is one spraying in it. All these zones can be created depending on the requirements to coating at the expense of location geometry of ASD, PGSM and substrate, slope angle of ASD and installation of additional reflecting screens on PGSM. After that the substrate goes to the zone of warehousing IV, where process of coating applying on it finishes.

The present method of coating allows to cure the process continuously, translocating substrates sequentially one after another from the packet through the working area. ASD and PGSM work in the continuous mode. After applying of coating to all substrates power supply and cooling of ASD, PGSM are switched off. Then the pumping-out system is switched off.

The installation works as follows.

First residual pressure 6,7 x 10^{-3} is set in the cabinet 1 with the help of the pumping-out system 2. Then power supply and rate of flux are switched on for ASD 9 and the discharge ignites, desirable anode – cathode voltage is set. The voltage is applied to PGSM 10 and cooling is switched on. The discharge in PGSM ignites. With the help of gas control in ASD optimum chamber pressure is set. The substrates travel mechanism 3 is switched on. The trip on the rack 7 works and grips the underpan with the substrate 6, located on the top of the packet. The underpans are set on rollers and move on guiding 5 of the duct 4. The duct 4 is set inside the cabinet. The drive device 8, containing the drive, transferres underpans from the charge zone I through the working area II to the zone of warehousing IV. During the traveling of one rack 7 together with the substrate 6 on the underpan from the charge zone 1 to the zone of warehousing IV the second rack 7 is reset and grips the following underpan with a substrate 6. The mechanism of a vertical displacement 12 transferres the frame 11 from ASD 9 and PGSM 10, providing distance between the cathode PGSM 10 and substrate 6 surface no more than 0.3 m. The following underpan with the substrate 6, having already past preliminary surface cleaning from impurities and surface preparation in the zone 1, moves through the working area II to the zone of warehousing IV. After treating of all substrates power supply and cooling of PGSM 10 are switched off. The pumping-out system 2 is switched off. Chamber pressure 1 is leveled with atmospheric pressure. Unloading and new charge of substrates 6 to the cabinet 1 are performed.

CLAIMS.

1. Method of metal coating applying on large-size substrates in vacuum, including cleaning of the substrate by inert gas ion beam and metal coating applying on the substrate by cathode spraying in the discharge in inert gas at simultaneous bombardment of the substrate by inert gas ion beam *distinguished* of subjects, that inert gas ion beam at cleaning is formed by the accelerator with the selfcontained electron drift and is guided towards moving substrate transferred on the keeper at the angle of 15-45° to substrate surface and cleaning and coating are performed at pressure (1,3-5,3) x 10^{-2} Pa and energy of inert gas ions in the range of 50-150 eV, and the coating starts not later ending of cleaning.

2. Vacuum installation for metal coating applying on large-size substrates containing the cabinet, pumping-out system, travel mechanism for substrates trimmed in a packet, plasma source of inert gas ions and generator of plasma superimposed metal *distinguished* of subjects, that the plasma source of inert gas ions is made as at any rate two accelerators with the selfcontained electron drift, with exit section directed towards to moving substrate at the angle of 15-45° to the plane of the substrate and set along a line, perpendicular to traffic direction of the substrate and parallel to the substrate, the generator of plasma of superimposed metal is made as the tubular cathode from superimposed metal and the tubular anode encompassing it on a spiral path electrically isolated from the cathode, joint with the source of a spark-over voltage and with a cooling system, thus the cathode is arranged parallel to the substrate, and the accelerator with the selfcontained electron drift and generator of plasma of superimposed metal are arranged sequentially in the traffic direction of the substrate and are set on a blanket frame, joint with the mechanism of a vertical displacement, the cathode of the generator of plasma of superimposed metal being set above a treated substrate apart no more than 0.300 m from its surface.

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(51) 6 C 23 C 14/34, C 03 C 17/06

Комитет Российской Федерации
по патентам и товарным знакам

(12) ОПИСАНИЕ ИЗОБРЕТЕНИЯ
к патенту Российской Федерации

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(56) 1. Отчет о НИР, № гос. регистрации 016 50072107, Калинин, КПИ, 1985, с.26-28. 2. Авторское свидетельство СССР № 159005, кл. С 03 В 17/06, 1963.

(54) СПОСОБ НАНЕСЕНИЯ МЕТАЛЛОСОДЕРЖАЩИХ ПОКРЫТИЙ НА КРУПНОРАЗМЕРНЫЕ ПОДЛОЖКИ В ВАКУУМЕ И УСТАНОВКА ДЛЯ ЕГО ОСУЩЕСТВЛЕНИЯ

(57) Использование: изобретение относится к оптическому приборостроению, лазерной технике, электронной и стекольной промышленности. Сущность изобретения: для пол-

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учения покрытий на крупноразмерных подложках в известном способе, включающем очистку подложки ионами инертного газа и нанесение покрытия при одновременной бомбардировке подложки пучком ионов, пучок ионов получают в ускорителе с замкнутым дрейфом, направляют на подложку под углом 15-45° к поверхности навстречу движущейся подложке при давлении $(1{,}3\text{-}5{,}3)\cdot10^{-2}$ Па и энергии ионов 50-150 эВ и начинают нанесение покрытия до окончания очистки. Вакуумная установка, работающая по данному способу, содержит, по крайней мере, два ускорителя с замкнутым дрейфом и генератор плазмы наносимого металла в виде трубчатого катода и охватывающего его спиралеобразного анода. 2 с.п. ф-лы, 3 ил.

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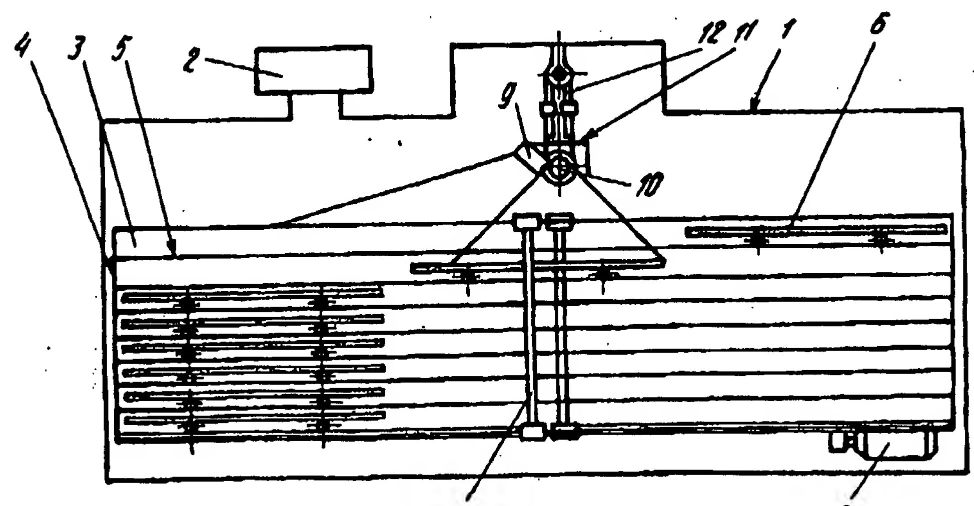
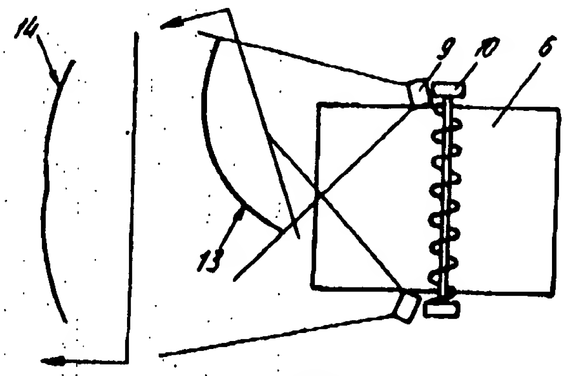
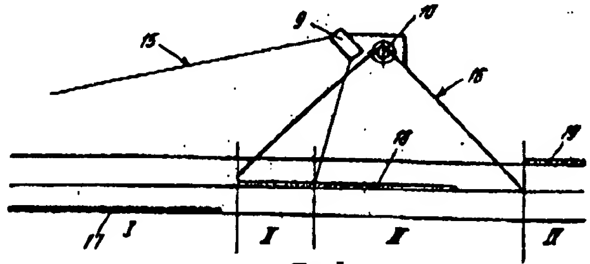


Фиг.1



Фиг.2



Фиг.3

Заказ 9п Подписное
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113834, ГСП, Москва, Раушская наб., 4/5

121873, Москва, Бережковская наб., 24 стр. 2.
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